N 72- 25544

NASA TECHNICAL MEMORANDUM

NASA TM X- 68053

NASA TM X- 68053

EFFECT OF TEMPERATURE ON THE TENSILE AND CREEP CHARACTERISTICS OF PRD49 FIBER/EPOXY COMPOSITES

by M. P. Hanson
Lewis Research Center
Cleveland, Ohio

TECHNICAL PAPER proposed for presentation at
Sixth Symposium on Composite Materials in
Engineering Design
St. Louis, Missouri, May 11-12, 1972

E-6903

EFFECT OF TEMPERATURE ON THE TENSILE AND CREEP CHARACTERISTICS OF PRD49 FIBER/EPOXY COMPOSITES

M. P. Hanson
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

Abstract

Tensile and creep data of PRD49-I and -III fiber/epoxy-resin composites are presented. Tensile data were obtained from 20 K (-423° F) to 477 K (400° F). Tensile strengths and moduli were determined at selected temperatures. Creep data up to 1000 hours are presented of fiber composites at 279 K (75° F), 422 K (300° F) and 450 K (350° F) at stress levels of approximately 50 and 80 percent of the ultimate tensile strength at 297 K (75° F). Details are presented of tensile specimens and test procedures used in the investigation.

INTRODUCTION

DuPont's recently developed organic fiber, PRD49, has been reported to have excellent reinforcing capabilities in structural composites.[1,2] The fiber has high tensile strength and modulus and a low density that yields specific properties that compare favorably to those of currently available high performance fibers such as graphite, boron, and glass. Results of short-term tensile tests have shown that the PRD49 fiber retains 75 percent of the room temperature tensile strength at 477 K (400° F) and a reduction of 18 percent in both the tensile modulus and elongation.[1] After 450 hours exposure at 514 K (465° F) in air, the fiber retains 70 percent of the room temperature tensile strength. The fiber has also shown low creep properties up to 1000 hours duration at room temperature.[1] Data on the creep characteristics of the fiber at elevated temperatures, however, are limited.

The purpose of the present investigation was to study the elevated temperature creep characteristics of unidirectional PRD49/epoxy composites at various stress levels. Tensile strength and modulus were also determined of the composites at temperatures ranging from 20 to 477 K (-423 to 400° F).

MATERIALS AND FABRICATION

PRD49 fiber types I and III were used in this investigation. Typical properties of the fibers are listed in table I. The yarn area in calculating the fiber stress was based on the weight per unit length divided by the fiber density. The resin matrix formulation[3] and cure cycle are also

TM X-68053

noted in table I. The formulation was chosen on the basis of its high-deformation temperature.

Before fabrication, the fiber was dried at 339 K (150° F) for 16 hours. Two types of specimens were used to investigate tensile and creep properties. Both specimens were made using a filament winding technique. Testing of the PRD49-1 was done with a loop specimen in which 100 turns of yarn were wound over two end-spools in three layers and impregnated with the epoxy resin. The resulting specimen consisted of two parallel bands with a known fiber count bonded to end spools in a manner similar to that shown in Fig. 1(a). The loop specimen was developed with the intent of minimizing the usual gripping problems encountered in testing a uniaxial fiber/resin composite tensile specimen. Although the loop concept proved adequate as a tensile and creep specimen at normal temperature, the specimen was found to be limited in performance at elevated temperatures.

Because of the limitation encountered with the loop specimen, a unidirectional-tensile specimen was developed for continued testing of PRD49-III composites. The fabrication details of this specimen was developed for continued testing of PRD49-III composites. The fabrication details of this specimen are shown in Fig. 1. The specimen was produced from a filament wound preform as shown in Fig. 1(a). It was reasoned that the filament-wound preform would provide in situ wound end reinforcement of the tensile specimen and also provide a reinforced area for the extensometer grips. Basically, the preform consists of three 1.27 cm by 25.4 cm (1/2 by 10-in.) unidirectional layers filament-wound about the end spools. As shown in Fig. 1(a), the outer fiber layers are separated from the middle layer by means of thin Teflon-coated glass-cloth separators which are placed between the individual layers during the winding process. The separators isolate the reinforced extensometer gripping area and test section from the end reinforcement. The center separator cloth approximately 5.08 cm (2.0-in.) long establishes the gage section of the final specimen. After curing the preform, the composite is removed from the end spools and sectioned into two separate flat strips approximately 22.8 cm (9.0-in.) long. The outer layers are carefully removed from the gage section area by saw cuts exposing the middle layer. Additional cuts are also made to release the extensometer gripping area from the end reinforcement as shown in Fig. 1(b). Further reinforcement of the tensile specimen ends is provided by aluminum tabs that are adhesively bonded to the unidirectional composite, Fig. 1(c). The aluminum tabs also provide a means of attaching pinned grips (fig. 1(d)) that adapt to either tensile or creep testing machines.

In addition to the in situ end reinforcements of the tensile specimen, the filament winding fabrication process also provides essentially perfect collimation of the fibers. Another advantage of the specimen is that no longitudinal cutting is required to establish the width of the specimen. Cutting unidirectional tensile specimens from flat laminates unavoidably results in some discontinuous fibers along the cut edges of the specimen.

APPARATUS AND PROCEDURE

TENSILE TESTS

Tensile tests were performed to establish tensile strength and modulus at various temperatures from 20 K (-423° F) to 477 K (400° F). Cryogenic tensile tests were conducted by immersion of the specimens in either LH_2 for 20 K (-423° F) tests or LN_2 for 77 K (-320° F) tests. Elevated temperature tests were performed in an environmental oven that was equipped with a thermocouple probe located in the proximity of the test section for temperature control. The tests were performed in about 15 minutes after the temperature had stabilized. All tests were run at a cross-head loading rate of 1.27 mm

(0.05-in.) per minute. Strain was measured at all temperatures by means of resistance strain gages that were adhesively bonded to the specimen test section. Tensile tests of PRD49-I were performed on the continuous loop specimens while PRD49-III were performed on the unidirectional tensile specimen described previously.

CREEP TESTS

Creep tests were performed in static loading machines equipped with lever arms that provided a 20:1 ratio of specimen load to dead weight load. The machines are provided with a heating chamber to maintain constant temperature throughout the duration of the test. The temperature was monitored by a thermocouple attached to the test section of the specimen. Creep tests were performed at 297 K (75^{o} F), 422 K (300^{o} F) and 450 K (350^{o} F). The stress levels were approximately 50 and 80 percent of the ultimate short-time tensile strength of the composite determined at the test temperatures.

Creep measurements were made of PRD49-I using loop specimens and of PRD49-III using the unidirectional tensile specimens. Creep values were obtained from loop specimens by applying a correction factor to the overall elongation of the specimen. The correction factor was determined from the ratio of the unit strain, measured by a strain gage, to the corresponding overall elongation measured during a tensile test.

The creep of PRD49-III was measured by means of a clamp-on-extensometer with the grips attached to the reinforced areas of the specimen as described in the Materials and Fabrication section. The grips were set at a 5.08 cm (2.0-in.) gage length. Deformation of the test section was transferred from the grips to the outside of the testing chamber by means of rod and tube extensions. A linear-variable differential transformer was located outside the chamber and attached to the rod and tube extensions. The calibrated output from the LVDT was traced on a strip chart recorder. The time was recorded by an accumulative counter that automatically shutoff upon failure of the specimen.

DISCUSSION AND RESULTS

TENSILE PROPERTIES

PRD49 fiber is a unique organic fiber in that the stress-strain relationship is linear up to its ultimate tensile strength. This behavior was observed for both type I and III fibers throughout the temperature range from 20 K (-423^{o} F) to 477 K (400^{o} F). Figs. 2 and 3 show the stress-strain relation at selected temperatures. In this investigation stress is presented in terms of fiber stress because of the precisely known fiber content wound into the loop and unidirectional tensile specimens. The fiber stress was determined by dividing the load by the total fiber area. The strength contributed by the resin can be considered negligible (about 1.0 percent) because of the high fiber to resin modulus ratio. The average fiber tensile strength values at 297 K (75^{o} F) was 221 000 N/cm^2 (320 000 psi) and 294 000 N/cm^2 (425 000 psi) for the type I and III fibers respectively. Corresponding tensile moduli were 16.7$\times 10^6$ N/cm^2 (24.2$\times 10^6$ psi) and 15.0$\times 10^6$ N/cm^2 (21.7$\times 10^6$ psi). Short-time tensile-strength at 477 K (400^{o} F) for the type III fiber was about 73 percent of the 297 K (75^{o} F) tensile strength. At 20 K (-423^{o} F) the strength was about 91 percent of the 297 K (75^{o} F) tensile strength. However, a 77 K (-320^{o} F) the tensile strength was lower than at either 297 K (75^{o} F) or 20 K (-423^{o} F). The fiber tensile modulus for the type III fiber ranged from 20.7$\times 10^6$ N/cm^2 (30.0$\times 10^6$ psi) at 20 K (-423^{o} F) to 12.4$\times 10^6$ N/cm^2 (19.5$\times 10^6$ psi) at 477 K (400^{o} F). Plots of the fiber tensile strength and modulus of type III as a function of temperature is shown in Fig. 4.

An interesting observation is that the moduli of

PRD49 fibers increase appreciably with decreasing test temperature. This is in contrast to inorganic fibers that do not exhibit any significant change in modulus with temperature. This modulus increase might be attributed to the fact that polymeric materials become extremely brittle and more rigid at cryogenic temperatures. In certain applications the higher cryogenic modulus would be advantageous. In strain-limited filament-wound pressure vessels with metallic liners or fiber-overwrapped metallic-pressure vessels, e.g., at cryogenic temperatures a higher stress could be realized from the fiber for a given strain.

CREEP PROPERTIES

The creep behavior of polymeric materials is an important factor to be considered when using polymers in structural applications. Generally, polymers exhibit creep properties that are markedly accelerated by elevated temperatures. Because PRD49 is a polymeric fiber, its long-term creep properties at ambient and elevated temperatures were investigated.

Initial creep studies were made of PRD49-I fiber on loop specimens described in the Materials and Fabrication section. The results are shown in fig. 5 where percent total strain is plotted as a function of time to 1000 hours. It is seen that at 297 K (75° F) and at a fiber stress of 172 500 N/cm^2 (250 000 psi) approximately 80 percent of the 297 K (75° F) ultimate tensile strength the specimen experiences an accelerated primary creep of about 0.1 percent within the first few hours after initial loading. This is followed by secondary creep which shows essentially a constant creep rate for the 1000 hour test duration. The secondary creep amounted to about 0.05 percent for the 1000 hour duration. At 422 K (300° F) and at a fiber stress of 96 500 N/cm^2 (140 000 psi) the creep behavior is similar to that at 297 K (75° F) with the exception of a higher secondary creep rate at the lower stress level. The total creep elongation was about 0.2 percent. Comparing the two tests, it appears that temperature influences the creep rate of PRD49 fibers. Attempts to load the loop specimens at higher stress levels at 422 K (300° F) resulted in fracture of the specimen within the first few hours of the test. The failure location was usually at the ends of the specimen where the fibers looped over the spool pieces. Creep tests using the loop specimen were discontinued. Creep studies on PRD49-III were performed with the unidirectional tensile specimen previously described.

Figure 6 shows the percent total strain as a function of time for PRD49-III. It is seen that the fiber at 297 K (75° F) sustained a tensile stress of 207 000 N/cm^2 (300 000 psi) (approximately 71 percent of the 297 K (75° F) ultimate tensile strength) for the 1000 hour test duration. The type III fiber appears to have similar creep behavior to that of type I fiber at 297 K (75° F). However, some variations in creep were noted at 297 K (75° F) during the 1000 hour duration. Although no systematic records were made of the humidity throughout the test, it was noted that creep increased during periods of high humidity and experienced some recovery during periods of lower humidity. At 422 K (300° F) and at a stress level of 124 300 N/cm^2 (180 000 psi) the one specimen failed in 460 hours and a second second specimen failed in 375 hours. In both specimens the failures were preceded by progressive fiber failure extending over a period of several hours. The progressive failure was accompanied by an apparent increase in strain as indicated in Fig. 6. Prior to the onset of fiber failure the creep behavior was similar to that of the 297 K (75° F) test. At 450 K (350° F) the maximum time to failure for the type III fiber was 54 hours at a stress level of 124 300 N/cm^2 (180 000 psi) and 93 hours at 103 500 N/cm^2 (150 000 psi).

SUMMARY OF RESULTS

The following results of strength and creep properties were obtained from an investigation of PRD49-I and III fiber in an epoxy matrix:

(1) The fiber tensile strength at 297 K (75° F) was 221 000 and 294 000 N/cm^2 (320 000 and 425 000 psi) for the type I and III, respectively. Corresponding tensile moduli were 16.7×10^{6} and 15.0×10^{6} N/cm^2 (24.2×10^{6} and 21.7×10^{6} psi).

(2) Short-time tensile-strength retention at 477 K (400° F) for the type III fiber was about 73 percent of the 297 K (75° F) tensile strength. At 20 K (-423° F) the strength retention was about 90 percent of the 297 K (75° F) tensile strength.

(3) The fiber tensile modulus for the type III fiber ranged from 20.7×10^{6} N/cm^2 (30×10^{6} psi) at 20 K (-423° F) to 13.4×10^{6} N/cm^2 (19.5×10^{6} psi) at 477 K (400° K).

(4) At 297 K (75° F) the type I and type III fiber sustained tensile stress of 173 000 and 207 000 N/cm^2 (250 000 and 300 000 psi), respectively, for 1000 hours without failure. For both fiber types, the total creep elongation was about 0.15 percent during the 1000 hour test.

(5) At 422 K (300° F) type I fiber sustained a 96 600 N/cm^2 (140 000 psi) tensile stress for 1000 hours without failure. Type III failed in 460 hours at a tensile stress of 124 000 N/cm^2 (180 000 psi). The total creep elongation of type I in the 1000 hour test at 422 K (300° F) was about 0.20 percent.

(6) At 450 K (350° F) the type III fiber at a tensile stress of 103 700 N/cm^2 (150 000 psi) failed in 93 hours; however, no excessive creep occurred until incipient progressive fiber failure concluded the test.

(7) Depending on the test conditions, both fiber types showed typical accelerated primary creep behavior within the initial 25 hours. This was followed by secondary creep that occurred at a much lower rate.

(8) At 297 K (75° F) humidity appeared to have a minor affect on the creep behavior of PRD49 fiber.

REFERENCES

1. Moore, J. W.: PRD-49-A New Organic High Modulus Reinforcing Fiber. Proceedings of the 27th Annual Technical Conference, Reinforced Plastics Composites Institute, Society of the Plastics Industry, Inc., 1972, Sect. 17-F, p. 1.

2. Hoggatt, J. T.: High Performance Filament Wound Composites for Pressure Vessel Applications. Space Shuttle Materials. Vol. 3 of National SAMPE Technical Conference. Society of Aerospace Material and Process Engineers, 1971, pp. 157-167.

3. Soldatos, A. C.; Burhans, A. S.; Eckstein, B. H.; and Spence, G. B.: High-Performance Epoxy/Graphite Fiber Composites. Modern Plastics, vol. 48, no. 12, Dec. 1971, pp. 62-64, 66.

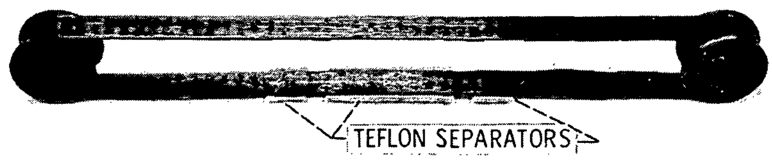
TABLE I. - TYPICAL PROPERTIES OF PRD49 FIBER

Fiber[a]	Specific gravity	Yarn area[b]		Tensile strength[c]		Tensile modulus[c]	
		cm^2	in.^{2}	N/cm^2	psi	N/cm^2	psi
PRD49-I	1.47	28.4×10^{-5}	4.4×10^{-5}	235×10^{3}	340×10^{3}	14.5×10^{6}	21.0×10^{6}
PRD49-III	1.45	32.3×10^{-5}	5.0×10^{-5}	277×10^{3}	400×10^{3}	13.1×10^{6}	19.0×10^{6}

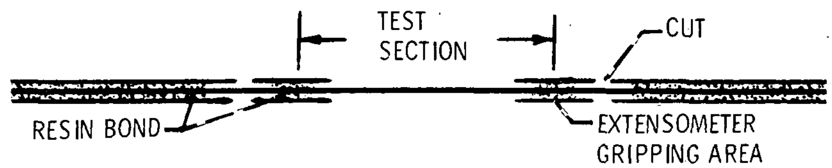
[a]E. I. DuPont de Nemours and Company
[b]Calculated cross-sectional area of yarn
[c]Typical properties reported by manufacturer at 297 K (75° F).

NOTE: Yarn impregnated with Union Carbide Corporation FRLB 4617 and Furane Plastics Inc., hardener 9247 (22 phr). Cure cycle: 2 hours at 355 K (180° F), 16 hours at 450 K (350° F).

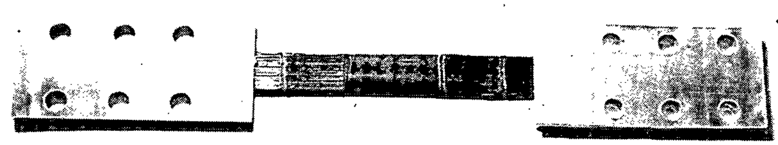
E-6903



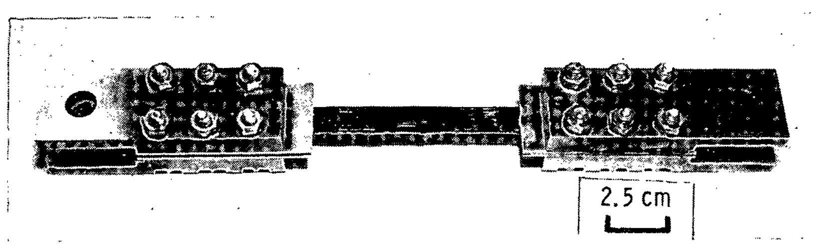
(a) THREE LAYER FILAMENT-WOUND SPECIMEN WITH TEFLON SEPARATORS.



(b) SCHEMATIC OF REMOVED OUTER FIBER REINFORCEMENT AT TEST SECTION.



(c) ALUMINUM TABBED TENSILE SPECIMEN SHOWING TEST SECTION AND EXTENSOMETER MOUNTING PADS.



(d) ASSEMBLED SPECIMEN WITH TENSILE GRIPS.

Figure 1. - Development of a filament-wound tensile specimen with in-situ end reinforcements.

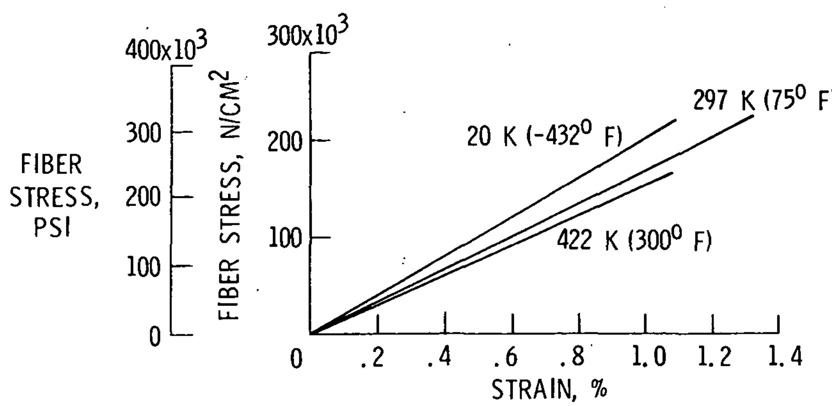


FIGURE 2. - TEMPERATURE EFFECT ON THE STRESS-STRAIN DIAGRAM OF PRD49-I/EPOXY COMPOSITE.

E-6903

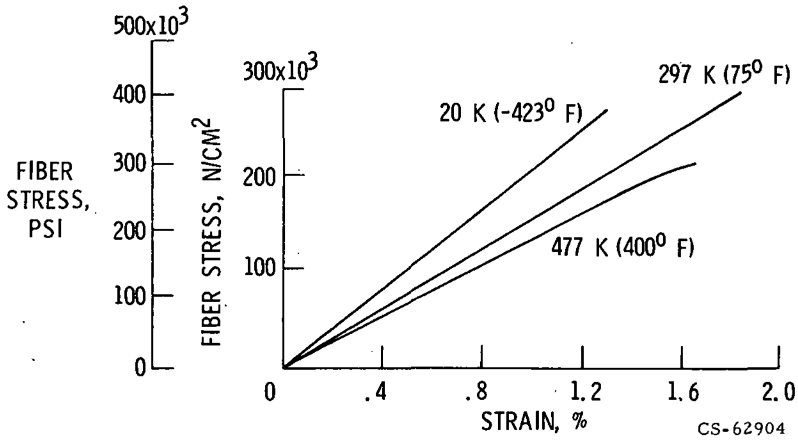


FIGURE 3. - TEMPERATURE EFFECT ON THE STRESS-STRAIN DIAGRAM OF PRD49-III/EPOXY COMPOSITE.

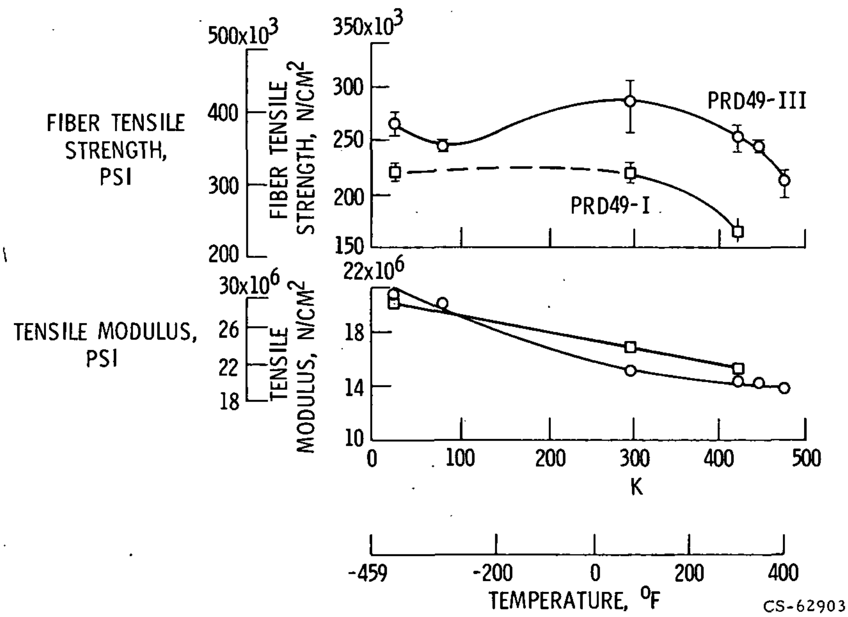


FIGURE 4. - FIBER TENSILE STRENGTH AND MODULUS OF PRD49-I AND PRD49-III/EPOXY COMPOSITES AS A FUNCTION OF TEMPERATURE.

E-6903

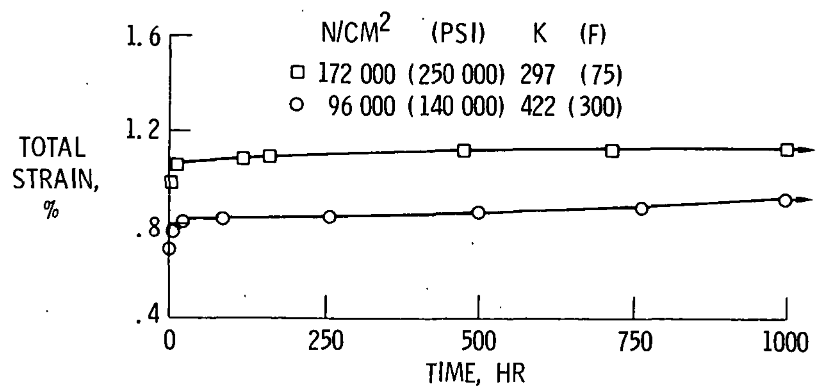


FIGURE 5. - CREEP-TIME RELATIONS FOR PRD49-I/EPOXY COMPOSITE AT VARIOUS STRESS AND TEMPERATURE LEVELS.

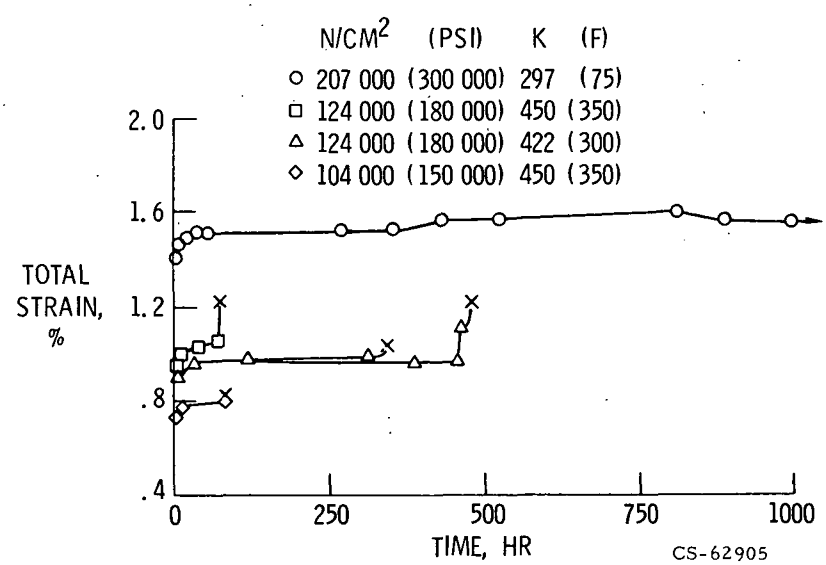


FIGURE 6. - CREEP-TIME RELATIONS FOR PRD49-III/EPOXY COMPOSITE AT VARIOUS STRESS AND TEMPERATURE LEVELS.